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**TITLE.** Preparation of  $AlB_4C$  Composites for Image Analysis

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## PREPARATION OF $B_4C$ /Al COMPOSITES FOR IMAGE ANALYSIS

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### ABSTRACT

Composites made by infiltrating  $B_4C$  networks with aluminum, or its alloys are of interest for lightweight armor applications. Image analysis plays an important part in correlating the microstructures of such composites with their mechanical properties. Accurate image analysis requires a high degree of perfection in the metallographic preparation, which is particularly difficult where the phases present have such disparate hardnesses and reactivities. Two preparation procedures have been developed that produce adequate contrast and definition for analysis of key microstructural features.

### INTRODUCTION

Composites of boron carbide and aluminum or aluminum alloys are currently of interest for lightweight armor applications.<sup>1</sup> These composites are made by infiltrating a porous, sintered skeleton of  $B_4C$  with liquid metal at temperatures high enough to promote wetting of the  $B_4C$ , but low enough to restrict undesirable reactions between them that produce other compounds.<sup>2-3</sup> For correlation with quasi-static and dynamic compression tests, soft shock recovery tests, and shock spallation experiments, samples of these composites were submitted for quantitative metallography.

A serious difficulty in preparing satisfactory metallographic sections of Al- $B_4C$  composites derives from the extreme disparity between their hardnesses, e. g. about 3200 HV for  $B_4C$ <sup>4</sup> and

160 HV for aluminum<sup>5</sup>. The objectives of the present work were to show the general features of the microstructures and to determine the volume fractions of the principal phases by image analysis. Initial attempts at sample preparation uncovered a number of problems with obtaining a sample suitable for image analysis.

## EXPERIMENTAL METHODS

Sixteen Al-B<sub>4</sub>C samples were prepared according to the methods outlined in Table 1. Image analysis samples were prepared by two methods. In the first method, the samples were used in the as-polished condition, with a white aluminum phase and a gray B<sub>4</sub>C phase. The second involved leaching out the aluminum phase with 10% NaOH for 5-20 minutes, rubbing the surface of the specimen with a black felt-tip marker pen to reduce the reflectivities of the internal pore surfaces, and removing the ink from the B<sub>4</sub>C phase by rubbing the specimen surface on a piece of paper. This resulted in dark areas where the aluminum phase had been, while the B<sub>4</sub>C was unaffected by the procedure and remained gray.

Image analysis was accomplished on a Dapple<sup>6</sup> image analyzer. Images were acquired from a video camera attached to a Leitz MM6 metallograph. Twenty areas were analyzed at 500X to determine the volume fraction of aluminum phase present in the composites.

## RESULTS AND DISCUSSION

### Initial Preparations and Difficulties

Initial preparations combined grinding the specimens on metal-bonded diamond discs (220 mesh through 15  $\mu$ m), polishing with a Whirlimot<sup>7</sup> attachment, an 8-inch wheel rotating at 233 rpm, Perforated Texmet as the polishing surface, and 3  $\mu$ m diamond abrasive with ethylene

glycol as the lubricant. In general, most samples prepared with Perforated Texmet show little polish relief. This procedure produced results that were satisfactory for most metallographic observations, but were found to be unsuitable for image analysis. Edge-rounding at the interfaces between the aluminum and the boron carbide introduced uncertainties in the determinations of accurate area percent values for the aluminum phase. This type of preparation is shown in Figure 1. In addition, discoloration of the aluminum phase was common, as shown in Figure 2, making setting of the gray levels to distinguish between aluminum and boron carbide difficult. Since both of these preparation defects introduce errors into the area percent measurements, an improved preparation technique was clearly needed.

In an effort to isolate and correct these problems, a number of grinding and polishing procedures were tried. The fact that the composites examined had different degrees of fineness and different volume fractions of aluminum introduced additional variables (see Table 1).

#### Grinding Media

In all cases, the total preparation time appeared to be controlled largely by the polishing time required to remove the damage introduced by the grinding operations. This damage was apparently produced by the tearing out of boron carbide particles. As shown in Table 1, use of Grid Abrade<sup>8</sup> grinding discs generally required the longest subsequent polishing times by a slight margin, perhaps because the holes in the discs reduced hydroplaning and allowed a more aggressive attack. Use of the metal-bonded diamond discs required somewhat shorter polishing times, but there was also evidence of severe grinding damage. The shortest polishing times were achieved when the grinding steps were performed entirely on silicon carbide papers. This is probably the result of a change in the grinding characteristics of these papers as they wear in during each stage of the grinding operation. In so doing, they may present surfaces that become progressively less aggressive as the wearing process proceeds. These papers, used with water as a lubricant, ranged from 120 to 600 grit and 600 soft.

### Polishing Media

The polishing media that are compared in Table I are: Perforated Texmet with 3  $\mu\text{m}$  diamond abrasive from an aerosol container, Texmet<sup>7</sup>, and silk, both with 3  $\mu\text{m}$  diamond paste. Preparations satisfactory for image analysis did not require polishing with diamond abrasives finer than 3  $\mu\text{m}$ . In addition, it did not appear to be necessary to use stainless steel wheels, using either kerosine or ethylene glycol, as is sometimes necessary to avoid deposition of copper on aluminum specimens. Samples polished on Texmet and Perforated Texmet had slightly rounded interfaces and the aluminum phase was often discolored. Usually, a specimen that had a discolored aluminum phase could be improved by a few more hours of polishing on silk. The best results were obtained using silk for the entire polishing operation. With this method, samples had little of the aluminum phase discoloration, minimal rounding at the phase boundaries, and required less time to achieve a sample preparation of sufficient quality for image analysis. Figures 3 and 4, show the quality attainable using silk as the final polishing surface. A common observation was that discoloration of the aluminum phase followed recharging of the polishing surface with diamond abrasive, either from an aerosol dispenser or from a paste syringe. It was also noted that once a satisfactory preparation had been achieved, samples subjected to additional polishing time sometimes developed discoloration of the aluminum phase again.

### Three Different Samples Prepared

Samples made from coarse (35  $\mu\text{m}$ )  $\text{B}_4\text{C}$ , Figure 4, required polishing times almost twice as long as those made from finer (5  $\mu\text{m}$ )  $\text{B}_4\text{C}$  powders, Figure 3. This is probably because the depth of damage due to pullout of the  $\text{B}_4\text{C}$  phase during grinding is directly related to the original particle size of the  $\text{B}_4\text{C}$ . For a given particle size in the  $\text{B}_4\text{C}$  powder used in making the

composites, the preparation time was not affected by the relative volume percents of the phases.

### Image Analysis

Perfection of the polish on the  $B_4C$  phase usually occurs much earlier than that of the aluminum phase. Therefore, it appeared logical to remove the aluminum phase from the polished surface, allowing the  $B_4C$  to be the lighter phase, relative to the pores formerly filled with the aluminum phase. This was accomplished by the sample preparation procedure previously outlined in the experimental methods section. A typical microstructure of a sample prepared by this method is shown in figure 5. Specimens prepared in this way can be processed more rapidly than by the preceding methods, but another difficulty is introduced. Small isolated islands of  $B_4C$  appearing in the aluminum phase may be unattached to the  $B_4C$  skeleton below the polished surface. These are removed along with the aluminum phase in the leaching process. This results in an area percent measurement that is higher than the true value for the aluminum phase. Such errors may be as high as 10% relative to the true value of the area percent of the aluminum phase for the samples that have been measured using both techniques. Another error that enters in is that the inherent porosity in the  $B_4C$  phase would be counted as aluminum using this technique. The porosity of most Al- $B_4C$  samples ranged from 0.5-2%.

The volume percent aluminum for the as-polished samples, 29.5%, and the leached and inked samples, 33.9%, were slightly lower than the 35 % aluminum suggested by the manufacturer. Unfortunately, it is difficult to confirm these results with elastic modulus data due to the inhomogeneity of this material and uncertainties about what model to use in calculating moduli.

### CONCLUSIONS

Of the methods tried, the most satisfactory surface preparation on Al- $B_4C$  composites for

image analysis were produced in the shortest times by grinding through 600 grit and 600 soft SiC papers, followed by polishing on silk with 3  $\mu\text{m}$  diamond abrasive with either kerosine or ethylene glycol as the lubricant. This minimizes relief between the major phases and produces a high quality polish suitable for image analysis. If both the porosity and the amount of unattached  $\text{B}_4\text{C}$  are low (<2% of the entire sample), then leaching out the aluminum followed by darkening of the internal pore surfaces with ink can produce a more rapid preparation for image analysis.

#### ACKNOWLEDGEMENTS

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5. Metals Handbook, vol. 2, 9th edition, Metals Park, Ohio, 1979, p. 715.
6. Dapple Systems, Sunnyvale, CA.
7. Buehler Ltd., Lake Bluff, IL.
8. TBW Industries, Inc., Furlong, PA

**TABLE I: SAMPLE PREPARATION**

	65% B <sub>4</sub> C (35 μ) - 35% Al												80% B <sub>4</sub> C (5 μ) - 20% Al		65% B <sub>4</sub> C (5 μ) - 35% Al	
	1	2	3	4	5	6	7	8	9	10	11	12	1	2	1	2
<b>GRINDING</b>																
Metal Bonded Diamond Disks	*		*	*		*							*		*	
Grid Abrade		*						*								
Silicon Carbide Papers					*		*		*	*	*	*		*		*
<b>POLISHING TIME (hours)</b> 3 μm diamond paste / spray																
Perforated Texmet	20	16	16							18	16		10		10	
Texmet				16												
Silk		2	2		10	10	10	10.5	7.5			14		4		4
<b>POLISHING WHEEL</b>																
Stainless Steel			*		*						*					
Bronze	*	*		*		*	*	*	*	*		*	*	*	*	*
<b>LUBRICANT</b>																
Kerosene			*							*		*		*	*	*
Ethylene / Propylene Glycol	*	*		*	*	*	*	*	*		*		*	*	*	*
<b>TOTAL POLISHING TIME (hours)</b>	20	18	18	16	10	10	10	10.5	7.5	18	16	14	10	4	10	4

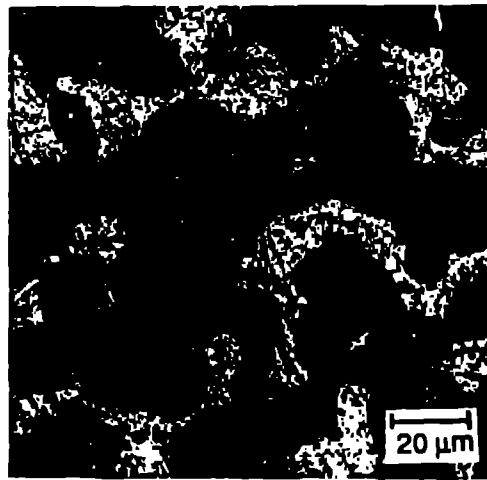


Figure 1. Edge-rounding at Al-B<sub>4</sub>C interfaces makes determination of interface position uncertain (500X).

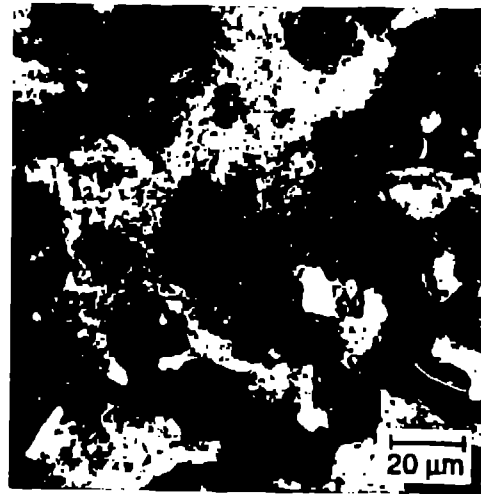


Figure 2. Discoloration of aluminum phase makes discrimination between Al and  $B_4C$  difficult (500X).

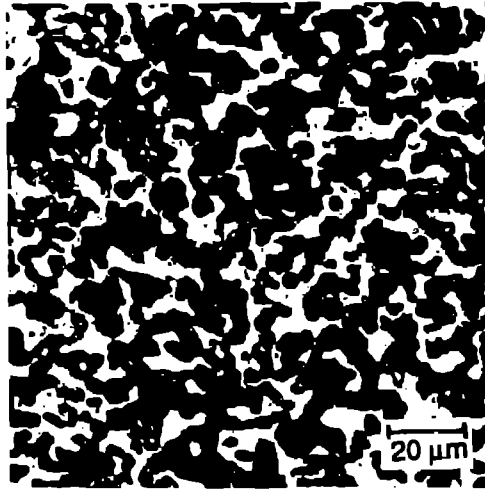


Figure 3. Satisfactory preparation of fine (5 μm particle size) Al-B<sub>4</sub>C composite (500X).

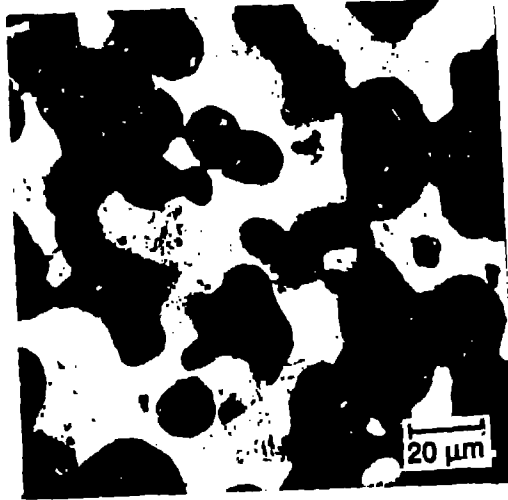
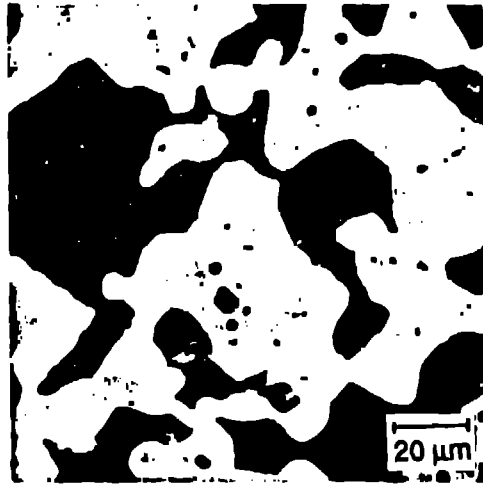


Figure 4. Satisfactory preparation of coarse (35  $\mu\text{m}$  particle size) Al-B<sub>4</sub>C composite (500X).



**Figure 5. Specimen preparation using leaching and inking method (500X).**

**PREPARATION OF AL-B<sub>4</sub>C COMPOSITES  
FOR IMAGE ANALYSIS**

**Presenter:**

**Ann Marie Kelly**

**Co-Authors:**

**Robert D. Reiswig**

**Mary Ann Hill**

**William R. Blumenthal**

## **INTRODUCTION**

- **Fabrication of Aluminum-Boron Carbide Composites**  
Sintered skeletal  $B_4C$  is infiltrated with aluminum or an aluminum alloy.
- **A large difference in hardness between the composite phases makes it difficult to obtain the high quality surface preparation required for image analysis.**
- **The volume % of the composite phases is critical in correlating structure and mechanical behavior. Image analysis is used to accurately determine the volume percent of the phases.**
- **In an effort to isolate and correct the problems extensive experiments were conducted with various grinding and polishing media, and a leaching and dyeing process.**

## METALLOGRAPHIC PROBLEMS LEADING TO IMAGE ANALYSIS ERROR



**ROUNDING**



**DISCOLORATION**

Both problems produce difficulties in choosing gray level settings to distinguish Aluminum and Boron Carbide phases.

## **EXPERIMENTAL ALTERNATE PREPARATION**

**Used for image analysis samples to avoid problems with discoloration of the Al phase and shorten preparation time**

### **PROCESS:**

**Aluminum phase was leached from the skeletal  $B_4C$  structure in 10% NaOH for 5 to 20 minutes**

**The leached pores colored with a black permanent marker, and excess ink was removed by rubbing the sample on paper**

# **EXPERIMENTAL GRINDING MEDIA**

**Grid Abrade**

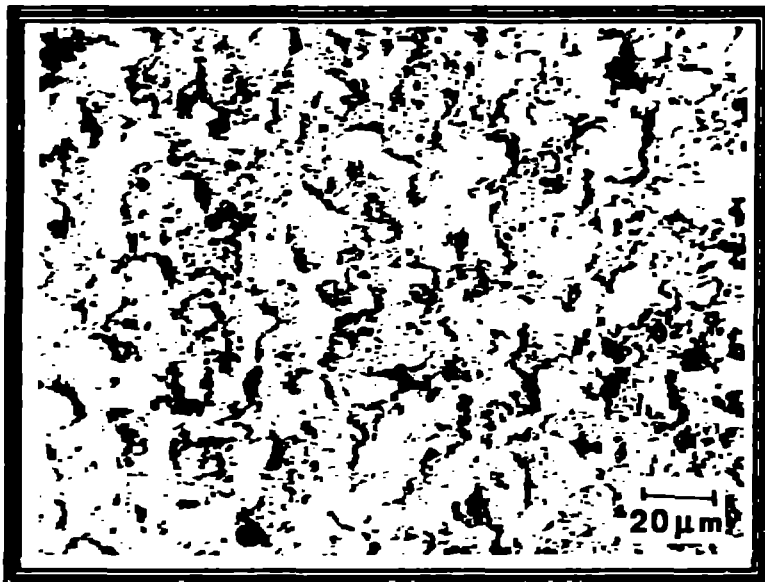
**Metal Bonded Diamond Disks**

**Silicon Carbide Papers**

## EFFECTS OF GRINDING MEDIUM ON Al-B<sub>4</sub>C COMPOSITES

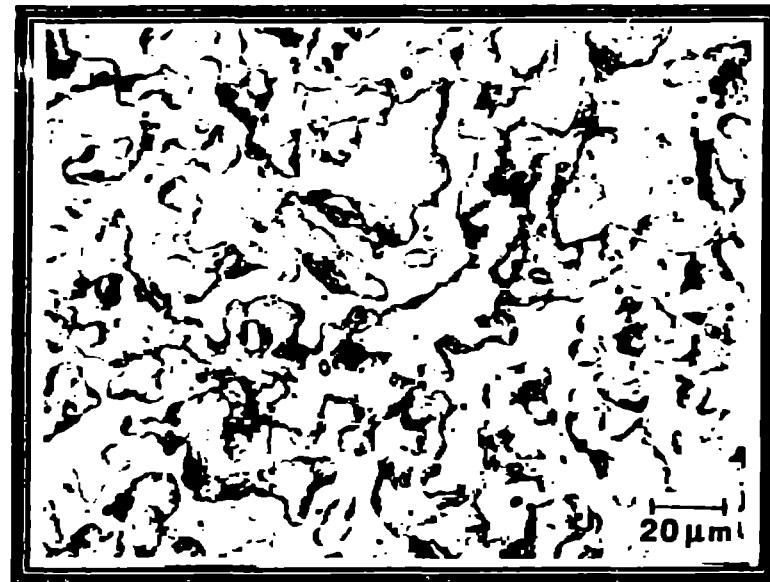
### Metal Bonded Diamond Disks

- Severe grinding damage in both Al and B<sub>4</sub>C phases
- Similar results were obtained using Grid Abrade



### Silicon Carbide Papers

- Less damage to the B<sub>4</sub>C phase
- Considerably less damage to Al phase



## EFFECTS OF GRINDING MEDIUM ON POLISHING TIME

Ground on  
Metal Bonded Diamond Disks

- Polishing time = 10 hours



Ground on  
Silicon Carbide Papers

- Polishing time = 7.5 hours



- Polishing Method:
- Silk
  - 3  $\mu$  diamond paste
  - Ethylene Glycol

# **EXPERIMENTAL POLISHING MEDIA**

## **Polishing Media**

3  $\mu$ m diamond spray/paste

**Perforated Texmet**

**Texmet**

**Nylon**

**Silk**

## **Polishing Wheels**

**Stainless Steel**

**Bronze**

## **Lubricants**

**Ethylene/Propylene Glycol**

**Kerosene**

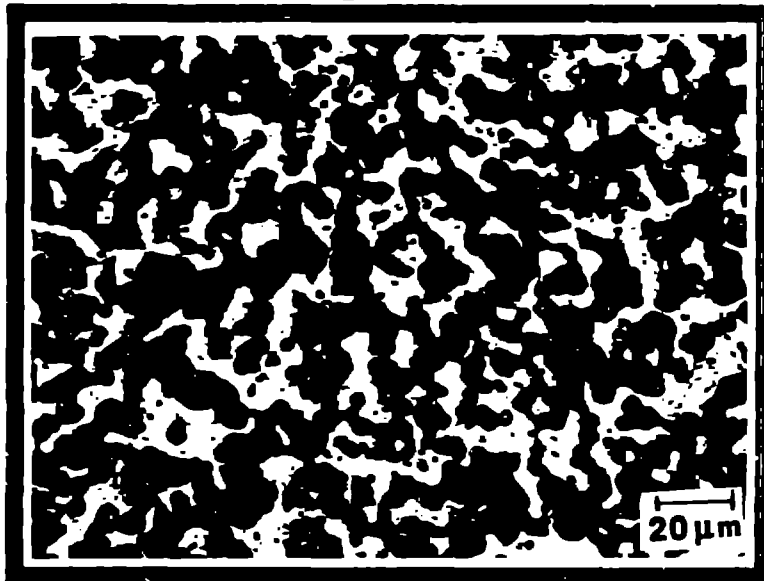
## **EFFECTS OF POLISHING MEDIA ON PREPARATION TIME AND QUALITY**

- **All media produced samples of sufficient quality for image analysis with the exception of nylon cloth.**
- **Dramatic reductions in preparation time are achieved with silk cloths.**

## EFFECTS OF B<sub>4</sub>C PARTICLE SIZE ON POLISHING TIME AND DEGREE OF DIFFICULTY

65% B<sub>4</sub>C-35% Al (5  $\mu$ )

- Sample preparation relatively simple
- Rounding and discoloration not observed
- Total polishing time = 4 hours



65% B<sub>4</sub>C - 35% Al (35  $\mu$ )

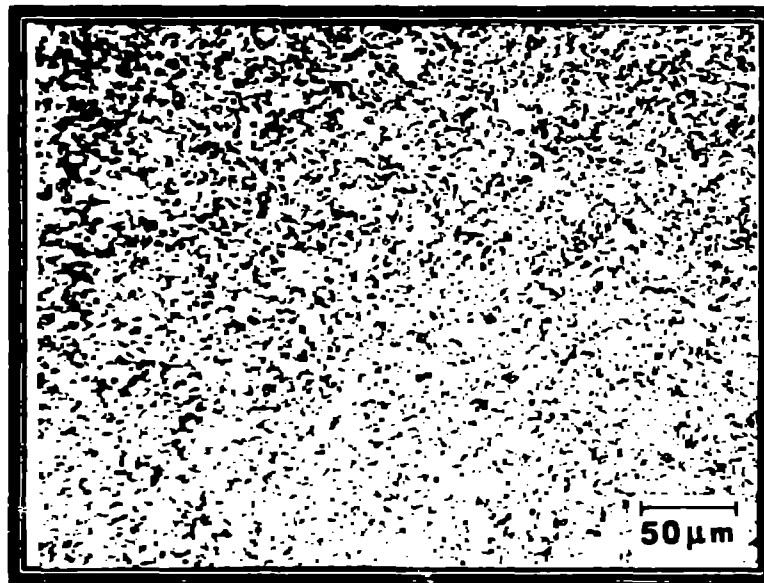
- Sample preparation very difficult
- Rounding and discoloration still a problem if under- or over-polished
- Total polishing time = 7.5 hours



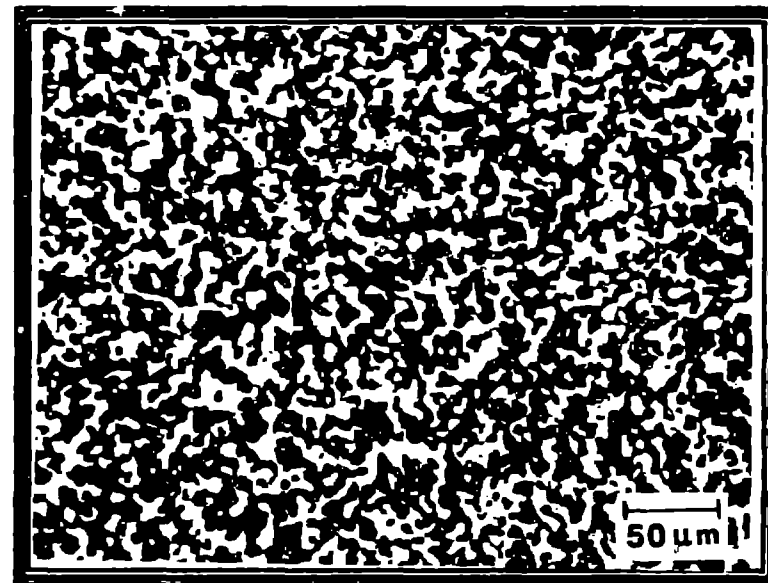
## EFFECTS OF VOLUME % $B_4C$ ON POLISHING TIME AND DEGREE OF DIFFICULTY

- Sample preparation relatively simple
- Rounding and discoloration not observed
- Total polishing time = 4 hours

80%  $B_4C$ -20% Al (5  $\mu$ )



65%  $B_4C$  - 35% Al (5  $\mu$ )



Samples were prepared on silk, with 3  $\mu$  diamond paste, and ethylene glycol.

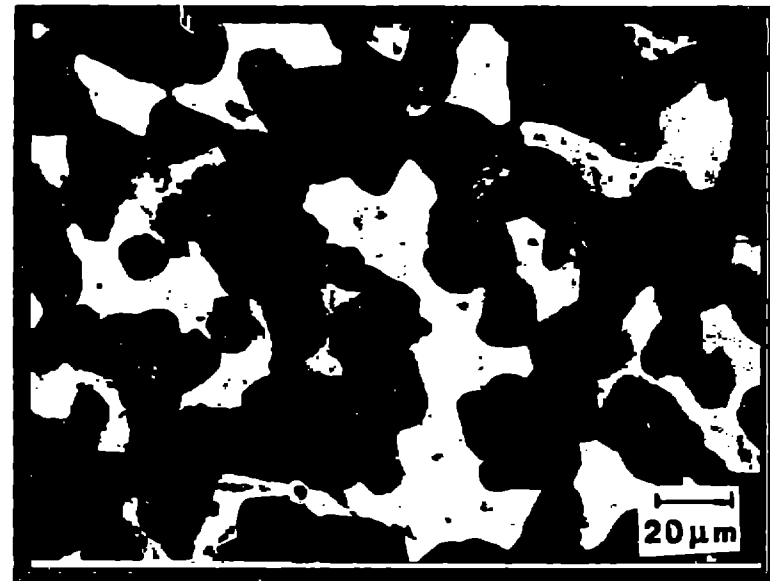
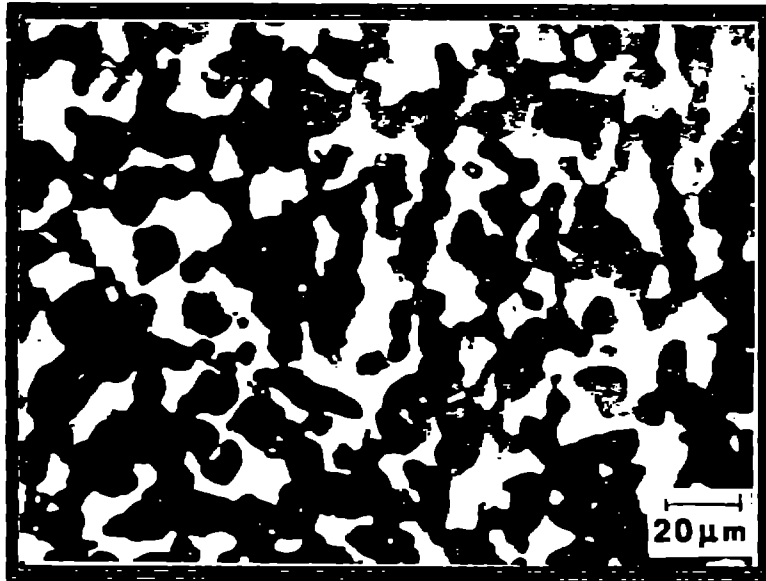
Samples polished on perforated Texmet achieved the same results, but took 10 hours.

**COMPARATIVE STUDY  
OF EXPERIMENTAL METHODS  
OF PREPARING SAMPLES FOR IMAGE ANALYSIS**

**Newly developed  
metallographic  
technique described by  
this paper**

**Alternate Preparation:  
Leaching aluminum phase  
from skeletal  $B_4C$   
structure, and dyeing  
aluminum background for  
contrast.**

## HIGHEST QUALITY SAMPLE PREPARATION TECHNIQUE



- Ground on Silicon Carbide Papers ( 120, 320, 600, 600 soft grit)
- Polished on silk
- 3  $\mu$  diamond paste
- Ethylene Glycol
- Equivalent results were achieved with bronze and stainless steel wheels

### New Metallographic Technique

#### Advantage:

- Very accurate image analysis of area % of aluminum phase

#### Disadvantage:

- Sample preparation is more time-consuming



### Aluminum phase leached & dyed

#### Advantage:

- Samples can be prepared relatively quickly

#### Disadvantage:

- Isolated islands of  $B_4C$  are removed during the leaching process, giving an artificially higher area % of the Al phase

